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(54) Title: IMPROVEMENTS IN OR RELATING TO PERFUME COMPOSITIONS

(57) Abstract: A perfume composition comprising a perfume component capable of inhibiting the production of odoriferous steroids by micro-organisms on the skin. The perfume component is capable of inhibiting the biotransformation of androstadienols to an-



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Title: Improvements in or relating to perfume compositions

Field of the Invention

This invention relates to perfume compositions, to products containing such perfume compositions, and to the use of a perfume component or perfume composition to deliver a deodorant effect. In particular, the invention relates to perfume components, mixtures thereof, and perfume compositions for reducing or preventing body malodour.

Background to the Invention

It is known that, at the point of secretion, sweat is odourless. Body malodour is the result of a variety of biotransformations of components of sweat by certain species of natural micro-organisms which live on the surface of the skin. These transformations produce a number of volatile odoriferous compounds such as steroidal compounds (e.g. 16-androstenes), amongst others, which contribute to body malodour.

There are three types of personal product routinely used to combat body malodour: perfumes, antiperspirants and deodorants. Products such as soaps, shower gels, body washes and laundry products are also intended to combat body malodour.

Perfumes may simply mask body malodour. However perfume compositions have been disclosed which exhibit a deodorant action. EP-B-3172, EP-A-5618, US-A-4304679, US-A-4322308, US-A-4278658, US-A-4134838, US-A-4288341 and US-A-4289641 all describe perfume compositions which exhibit a deodorant action when applied to human skin or when included in a laundry product used to launder textiles.

Antiperspirants work by blocking the sweat glands, thereby reducing perspiration.

Antimicrobial agents used in deodorants are designed to reduce the population, inhibit the



growth or diminish the metabolic activities of micro-organisms living on the surface of the skin. Typical agents of this nature include ethanol and Triclosan (2',4,4'-trichloro-2-hydroxydiphenyl ether) which are well known to exert antimicrobial effects. The use of common deodorant actives results in a non-selective antimicrobial action exerted upon most of the skin's natural microflora. This is an undesirable disadvantage of such deodorant formulations, since the natural microflora provides a protective barrier (colonisation resistance) against invasion by potentially pathogenic bacteria.

US-A-5643559 (Colgate-Palmolive Company) discloses deodorant active materials having an effective amount of  $Zn^{2+}$  ions for inhibiting bacterial exoenzymes responsible for the production of axillary malodour. The bacterial exoenzymes are further characterised as aryl sulphatase or beta glucuronidase.

DE-4343265 (Henkel) describes deodorant compositions comprising saturated dioic acid (C3-C10) esters. The active inhibits a sweat decomposing esterase and the compositions are said not to disturb the skin's natural microflora.

WO 94/07837 (Unichema) describes certain novel unsaturated dioic acids having between 8 and 22 carbon atoms. The potential use of these acids to treat malodour is also described.

Gower et al. (*J. Steroid Biochem. Molec. Biol.*, (1994) Vol. 48, No. 4, pp 409-418) discloses the importance of certain bacterial enzymes involved in bacterial steroid metabolism in the production of odoriferous steroids, and proposes a series of interconversions between some of these metabolites.

Talalay, P.: Hydroxysteroid Dehydrogenases in *The Enzymes, VII*, 2nd Ed., (Boyer, P., Lardy, H., and Myrback, K., eds.), Academic Press, NY, 177, 1963, describes that 3[[alpha]] hydroxysteroid dehydrogenase from *Pseudomonas testosteroni* is inhibited by heavy metals and sulfhydryl-binding reducing agents.

Nakajin et al. (J. Steroid Biochem. Molec. Biol., (1991) Jan;38(1):95-9) discloses that the -conazole antifungal agents have a mode of action based on the inhibition of sterol metabolism. The activity of the enzyme (16-ene-C19-steroid synthesizing enzyme) responsible for the conversion of C21-steroids to 16-ene-C19-steroids, which was localized on pig testicular microsomes, was inhibited by some typical imidazole antifungal compounds such as clotrimazole, econazole, miconazole and ketoconazole which are known to be universal inhibitors of cytochrome P-450 dependent enzymes.

Lavallee et al. (*J. Steroid Biochem. Molec. Biol.* (1993) Jul;46(1):73-83) describes 20 beta-hydroxypregnenolone as a more potent inhibitor of 5,16-androstadien-3 beta-ol synthetase than of 17-hydroxylase and for the latter enzyme activity, the Ki(app) was lower than that for 17-hydroxypregnenolone itself.

Watabe et al. (*J. Biol. Chem.* (1985) Jul 25;260(15):8716-20) describes that the C16-double bond of the steroid androsta-5,16-dien-3 beta-ol, is oxidized by male rat liver microsomes to 16 alpha,17 alpha-epoxyandrost-5-en-3 beta-ol; 16 beta,17 beta-epoxyandrost-5-en-3 beta-ol; androst-5-ene-3 beta,16 alpha,17 beta-triol; and androst-5-ene-3 beta,16 beta,17 alpha-triol, and this transformation is strongly inhibited with CO.

WO 00/01355 and WO 00/01358 describe agents useful in preventing or reducing body malodour by inhibiting the production of odoriferous steroids, wherein the agents inhibit the bacterial enzymes, bacterial 4-ene reductase and/or 5  $\alpha$ -reductase. Examples of active agents are described as dicarboxylic acids, phenyl compounds, monoterpene derivatives, sterols, flavonoids, steryl esters, 2,7-napthalenediol and oxyquinoline (WO 00/01355), and certain perfume components (WO 00/01358).

Several steroids, notably  $5\alpha$ -androst-16-en-3-one ( $5\alpha$ -androstenone),  $5\alpha$ -androst-16-en- $3\alpha$ -ol ( $3\alpha$ -androstenol) and androsta-4, 16-dien-3-one (androstadienone) are known to be highly odorous in the context of human axillary odour. The biotransformations effected by a micro-organism on the components of sweat to produce such odoriferous products or

intermediates, occur via a number of possible, and typically, ill-defined metabolic pathways.

It has been suggested in the prior art (Gower et al) that odorous steroids, e.g. androstenones, are formed by the biotransformation of typically non-odorous steroids i.e. steroids present at levels below the threshold of human olfactory detection, by the action of micro-organisms present on the skin surface. More particularly,  $5\alpha$ -androsta-5, 16-dien- $3\beta$ -ol (androstadienol) was a source of the odorous androstenones.

#### Summary of the Invention

The present invention is based on extensive testing of perfume components to determine whether a particular component is capable of inhibiting the biotransformation of androstadienols to androstenones, particularly  $5\alpha$ -androst-16-en-3-one, and thus is capable of inhibiting the production of odoriferous steroids by micro-organisms on the skin surface. Based on this testing, perfume components were identified, which whilst known, possess hitherto unappreciated deodorant properties. The invention thus enables perfume compositions to be defined that reduce or prevent body malodour.

Accordingly, in one aspect, the present invention provides a perfume composition comprising at least 30% by weight of one or more of the following perfume components; Armoise Tunisian oil, para-tert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, methylphenyl)propionamide, 2-ethyl-N-methyl-N-(3ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, methylphenyl)butanamide, dihydromyrcenol, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, alpha-terpineol, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3octahydro-2H-chromen-2-one, cis-4gamma-nonalactone, trimethyl-2-norbornanol, decenal, 3-(3-isopropylphenyl)butanal.

pine American oil;

The following perfume components are useful in the perfume compositions defined herein:

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Armoise Tunisian oil;
para-tert.butylphenylacetonitrile (also known as 'Marenil' where MARENIL is a trade
mark of Quest International);
dihydrolinalol (3,7-dimethyloct-6-en-3-ol);
N-ethyl-N-(3-methylphenyl)propionamide (also known as 'Agarbois' where AGARBOIS is
a trade mark of Quest International);
4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol;
ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate;
2-ethyl-N-methyl-N-(3-methylphenyl)butanamide (also known as 'Paradisamide' where
PARADISAMIDE is a trade mark of Quest International);
dihydromyrcenol (2,6-dimethyl-7-octen-2-ol);
(4-isopropylcyclohexyl)methanol;
3-methyl-5-phenylpentan-1-ol (also known as 'Mefrosol' where MEFROSOL is a trade
mark of Quest International);
2,2,2-trichloro-1-phenylethyl acetate (also known as Rosacetone or Roseacetone);
isobornyl acetate;
allyl amyl glycolate ('2-methylbutyloxyacetic acid, 2-propenyl ester');
alpha-terpineol;
acetyl cedrene (also known as 'Lixetone' where LIXETONE is a trade mark of Quest
International);
 tetrahydrogeraniol;
 citronellal;
 cuminic aldehyde (para-isopropylbenzaldehyde);
 cis-jasmone;
 methyl octyl acetaldehyde (2-methyldecenal);
 gamma-octalactone (5-butyldihydrofuran-2(3H)-one);
 octyl acetate;
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peppermint (Chinese);

- 1,3,3-trimethyl-2-norbornanyl acetate (fenchyl acetate);
- 1,3,3-trimethyl-2-norbornanol (fenchyl alcohol);

gamma-nonalactone;

octahydro-2H-chromen-2-one (also known as 'Octahydrocoumarin' where OCTAHYDROCOUMARIN is a trade mark of Quest International);

cis-4-decenal;

3-(3-isopropylphenyl)butanal.

The term "perfume component" is used herein to represent a material which is added to a perfume composition to contribute to the olfactive properties of the composition. A perfume component can be acceptably employed to provide odour contributions to the overall hedonic performance of products. Typically, a perfume component will be generally recognised as possessing odours in its own right, will be relatively volatile and often has a molecular weight within the range 100 to 300. Typical materials which are perfume components are described in "Perfume and Flavour Chemicals", Volumes I and II (Steffan Arctander, 1969).

For the purposes of the present invention, by perfume composition is meant a mixture of individual perfume components, and optionally one or more suitable diluents, which is used to impart a desired odour to the skin and/or product for which an agreeable odour is indispensable or desirable. Commonly used diluents are benzyl benzoate, diethyl phthalate, dipropylene glycol and isopropyl myristate. The concentration of perfume components referred to herein is relative to the total concentration of perfume components present in the composition, i.e. excludes any diluents.

To deliver high deodorant effects the perfume component(s) are preferably present in a perfume composition in an amount of 40% by weight of the total weight of the perfume composition, more preferably at least 45%, and most preferably at least 60%.

Additionally, or alternatively, a perfume composition in accordance with the present invention preferably comprises at least 3, more preferably at least 5, and even more preferably at least 10 of the specified perfume components.

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Thus, in a further aspect, the present invention provides a perfume composition comprising at least 3 of the following perfume components; Armoise Tunisian oil, paratert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4ethyltricyclo[5.2.1.0{2,6}]decane-2-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, 2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, dihydromyrcenol, carboxylate, 2,2,2-trichloro-1-3-methyl-5-phenylpentan-1-ol, isopropylcyclohexyl)methanol, phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, alpha-terpineol, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal.

The perfume components useful herein in a perfume composition may be incorporated into deodorant products which include, but are not limited to, body deodorants and antiperspirants including roll ons, sprays, gel products, stick deodorants, antiperspirants, shampoos, soaps, shower gels, talcum powder, hand creams, skin conditioners, sunscreens, sun tan lotions, and hair conditioners.

Thus, in an even further aspect, the present invention provides a deodorant product comprising a perfume composition in accordance with the invention.

A deodorant product preferably comprises at least 0.05% to 4%, more preferably 0.1% to 2% of a perfume composition by weight of the deodorant product.

The perfume components useful herein may also be conveniently employed for deodorant purposes by incorporation into other products, e.g. laundry and household products such as rinse conditioners, household cleaners and detergent cleaners. The perfume components

can be incorporated into textiles themselves during their production using techniques known in the art, to provide deodorant protection.

In a preferred embodiment of the present invention, an Odour Reduction Value, measured in human axillae as described in Example 4, of at least 10%, more preferably at least 30%, and particularly at least 45% is obtained.

One or more of the perfume components useful herein may be mixed with other perfume components, e.g. perfume components of the prior art having deodorant properties, to formulate perfume compositions with desired deodorant and hedonistic properties.

In one such embodiment, there is provided a perfume composition as defined herein, wherein the perfume composition additionally comprises at least 15% by weight, preferably at least 30% by weight, of one or more of the following perfume components: acetyl di-iso-amylene, acetyl tributyl citrate, aldehyde C10 (i.e. decenal), Amber AB 358 (available from Quest International), amyl salicylate, anisyl acetate, Azarbre\*, benzyl salicylate, cis-3-hexenyl salicylate, citral, citronellol, clove leaf distilled, coriander, cyclamen aldehyde, decen-1-ol, dihydroeugenol, diphenylmethane, Dupical\*, Empetaal\*, geraniol, helional i.e. 2-methyl-3-(3,4-methylene-dioxyphenyl)propanal), Ionones (alphaand beta-), Jasmacyclene\*, 3-(4-methyl-4-hydroxyamyl)-3-cyclohexene carboxaldehyde, methyl eugenol, methyl isoeugenol, Ortholate\*, para-cresyl methyl ether, 2-phenylethyl alcohol, para tert. butyl cyclohexyl acetate, rose oxide (racemic), styrallyl acetate, tetrahydrolinalol, and vanillin; wherein all asterisked materials are trade marks of Quest International.

In a preferred embodiment, there is provided a perfume composition comprising:

(i) at least 30% by weight of the perfume composition of at least 3 of the following perfume components: N-ethyl-N-(3-methylphenyl)propionamide, 2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, dihydromyrcenol, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, alpha-terpineol, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic

aldehyde, cis-jasmone, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal; and

(ii) at least 30% by weight of the perfume composition of one or more of the following perfume components: acetyl di-iso-amylene, acetyl tributyl citrate, aldehyde C10, Amber AB 358, amyl salicylate, anisyl acetate, Azarbre, benzyl salicylate, cis-3-hexenyl salicylate, citral, citronellol, clove leaf distilled, coriander, cyclamen aldehyde, decen-1-ol, dihydroeugenol, diphenylmethane, Dupical, Empetaal, geraniol, helional, alpha-ionone, beta-ionone, Jasmacyclene, 3-(4-methyl-4-hydroxyamyl)-3-cyclohexene carboxaldehyde, methyl eugenol, methyl isoeugenol, Ortholate, para-cresyl methyl ether, 2-phenylethyl alcohol, para tert, butyl cyclohexyl acetate, rose oxide, styrallyl acetate, tetrahydrolinalol, and vanillin.

Also included within the scope of the invention is a method, particularly a cosmetic method, for reducing or preventing body malodour by topically applying to human skin a composition comprising a perfume component selected from at least one of the following; Armoise Tunisian oil, para-tert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, methylphenyl)propionamide, 2-ethyl-N-methyl-N-(3ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, methylphenyl)butanamide, dihydromyrcenol, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, alpha-terpineol, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3octahydro-2H-chromen-2-one, cis-4gamma-nonalactone, trimethyl-2-norbornanol, decenal, 3-(3-isopropylphenyl)butanal.

Preferably, the composition is a perfume composition.

Preferred perfume components for use in the method as defined above are selected from one or more of the following; Armoise Tunisian oil, para-tert butylphenylacetonitrile,

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dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, 2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal.

The method thus comprises topically applying to human skin, one or more of the specified perfume components which is(are) capable of reducing or preventing body malodour by inhibiting the production of odoriferous steroids by micro-organisms present on the skin surface, wherein the perfume component is capable of inhibiting the biotransformation of androstadienols to androstenones. Typically, the specified perfume components inhibit the production of odoriferous steroids by Coryneform bacteria present on the skin surface, particularly Corynebacterium spp. The inhibitory effect of the perfume components useful herein can be achieved antimicrobially or sub-lethally.

The antimicrobial effects of compounds, e.g. perfume components, are usually divided into two types; they can either inhibit bacterial growth (bacteriostatic action) or alternatively they can act by directly killing existing viable bacteria (bactericidal action).

The bacteriostatic action of a compound "X" such as a perfume component, can be tested for *in vitro* by inoculating a standard, small number of bacteria into broths containing an appropriate range of concentrations of X. The broths are then incubated for a suitable time, and growth compared with a control containing no inhibitor. The broth containing the lowest concentration of X which shows reduction of growth compared to the control broth is defined as the minimum inhibitory concentration (MIC).

The determination of bactericidal action of a compound "Y" such as a perfume component is carried out by adding various concentrations of compound Y to replicate broths

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containing relatively high, standard numbers of bacteria. After a certain period allowing any antibacterial activity to take place, aliquots of the bacterial cultures are diluted (usually in 10-fold steps) and dispensed onto agar plates. The plates are incubated with the expectation that each viable cell should produce a visible colony. The numbers of colonies are multiplied to take account of the dilution, to establish the number of viable cells in the broths. Once again, the broths containing compound Y are compared with an untreated control broth. The minimum concentration of compound Y which causes a reduction in the viable number of bacteria is the minimum bactericidal concentration (MBC). MBC can also be expressed in terms of the MBC required to produce a certain degree of killing (for example, a 3 log<sub>10</sub> reduction in count, equivalent to a 99.9% kill). Still further, the MBC can be expressed in kinetic terms - the time of exposure to an agent required for a given MBC effect.

A further possibility is that the process of inhibition could be sub-lethal (or sub-MIC), whereby the perfume components interfere with the metabolic process, but typically do not inhibit bacterial growth.

Preferably, the bacterial production of odoriferous steroids is reduced by at least 50%, more preferably by at least 70%, particularly by at least 80%, and especially by at least 90%. Three modes of achieving a reduction of odoriferous steroid production are possible. In the first mode, the perfume components (or perfume compositions) may act by direct (overt antimicrobial) killing of skin bacteria, e.g. by more than 10-fold; in the second mode, they may act on odoriferous steroid generation whilst maintaining a microbial cell viability of at least 70%; in the third mode, they may inhibit odoriferous steroid generation, at a concentration below the minimum inhibitory concentration (MIC), determined as described in Example 1 below. The third mode is preferred, since this provides malodour counteraction benefits, whilst leaving the natural skin microflora undisturbed. Thus, preferably the bacterial production of odoriferous steroids can be reduced or eliminated without significantly disturbing the skin's natural microflora. This may be achieved by inhibiting the bacterial enzymes responsible for the production of odoriferous steroids, in particular the androstenones such as  $5\alpha$ -androst-16-en-3-one.

In an even further aspect the present invention provides use of one or more of the following perfume components; Armoise Tunisian oil, para-tert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, 2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal, as a deodorant active.

In a still further aspect the present invention provides use of one or more of the following para-tert.butylphenylacetonitrile, Armoise Tunisian oil, components; perfume dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, 2-ethyl-N-methyl-N-(3methylphenyl)butanamide, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, 3-(3-isopropylphenyl)butanal, in the cis-4-decenal, octahydro-2H-chromen-2-one, manufacture of a composition for reducing or preventing body malodour.

Based on the MIC value evaluated for a particular perfume component, it is possible to select and combine those perfume components having low MIC values which are likely to be antimicrobially active, and to formulate a deodorant product which has some degree of anti-microbial activity. An example of this is a product including an antimicrobially effective amount, typically between 0.05% and 4% by weight, preferably between 0.1% and 2% by weight, more preferably between 0.5% and 1.5% by weight, of a perfume

composition comprising at least 30% by weight of one or more of the following perfume components:

N-ethyl-N-(3-methylphenyl)propionamide; 3-methyl-5-phenylpentan-1-ol; 2,2,2-trichloro-1-phenylethyl acetate; pine American oil; cis-4-decenal; 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol; cuminic aldehyde; methyl octyl acetaldehyde; Armoise Tunisian oil; dihydromyrcenol; allyl amyl glycolate; alpha-terpineol; cis-jasmone; peppermint (Chinese); gamma-nonalactone; octahydro-2H-chromen-2-one; para-tert.butylphenylacetonitrile; dihydrolinalol; tetrahydrogeraniol; and 1,3,3-trimethyl-2-norbornanol;

optionally in combination with perfume components having known high antimicrobial activity such as phenylethyl alcohol, geraniol, cinnamic acid, benzyl alcohol, and citral.

Likewise, it is also possible to select and combine those perfume components with higher MIC values which are likely to sub-lethally inhibit odoriferous steroid generation, and to formulate a deodorant product with minimal antimicrobial activity. Such a product may include, for example, appropriate levels of a perfume composition, typically between

0.05% and 4% by weight of the deodorant product of a perfume composition, preferably between 0.1% and 2% by weight, more preferably between 0.5% and 1.5% by weight, the perfume composition comprising at least 30% by weight of one or more of the following perfume components:

ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate;
2-ethyl-N-methyl-N-(3-methylphenyl)butanamide;
(4-isopropylcyclohexyl)methanol;
isobornyl acetate;
acetyl cedrene;
citronellal;
gamma-octalactone;
octyl acetate;
1,3,3-trimethyl-2-norbornanyl acetate;
3-(3-isopropylphenyl)butanal.

The invention also provides the use of a perfume component to inhibit the biotransformation of androstadienols to androstenones, in particular the biotransformation of androsta-5,16-dien-3 $\beta$ -ol to  $5\alpha$ -androst-16-en-3-one.

The invention further provides the use of a perfume composition, comprising at least 30% by weight of one or more perfume components capable of inhibiting the biotransformation of androstadienols to androstenones, to reduce body malodour.

The invention further provides the use of a deodorant product, comprising a perfume component, to reduce body malodour by inhibiting the biotransformation of androstadienols to androstenones.

The invention still further provides a method of producing a perfume composition which comprises (i) evaluating perfume components on the ability to inhibit the biotransformation of androstadienols to androstenones, (ii) selecting perfume components on the ability to

inhibit the biotransformation of androstadienols to androstenones, and (iii) mixing together two or more of said selected perfume components, optionally with other perfume components.

The invention still further provides use of a perfume composition comprising a perfume component to reduce body malodour, characterised in that the composition comprises at least 30% by weight of at least one of the perfume components specified in the paragraph bridging pages 5 and 6 above.

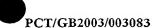
The invention is illustrated by the following examples.

## Example 1: Minimum Inhibitory Concentration (MIC)

The minimum inhibitory concentration of a perfume component was determined by the following method.

A culture of the test strain - Corynebacterium xerosis NCTC 7243 (National Collection of Type Cultures, Public Health Laboratory Service, Central Public Health Laboratory, 61 Colindale Avenue, London, NW9 5HT) was grown in 100ml of tryptone soya broth (TSB) (Oxoid, Basingstoke, UK) for 16-24 hours, in a shaken flask at 37°C. The culture was then diluted in sterile 0.1% TSB (Oxoid, Basingstoke, UK) to give a concentration of bacteria of approximately 10<sup>6</sup> colony forming units (cfu) per ml.

Perfume or perfume component samples were diluted in sterile TSB to give stock solutions with final concentrations of 40,000 ppm (perfume) or 20,000 ppm (perfume component). Each row of a standard, 96-well plastic microtitre plate (labelled A-H) was allocated to one sample, thus eight samples per plate. Row H contained only TSB for use as a bacterial control to indicate the degree of turbidity resulting from bacterial growth in the absence of any test material. Aseptically, 200µl of the initial dilution of perfume/perfume component was transferred to the 1<sup>st</sup> and 7<sup>th</sup> well of the appropriate row. All other test wells were filled with 100µl of sterile TSB using an 8-channel micro-pipette. The contents of each of



the wells in column 1 were mixed by sucking samples up and down in pipette tips, before 100µl was transferred to column 2. The same sterile pipette tips were used to transfer 100µl of each well in column 7, into the appropriate well in column 8. This set of eight tips was then discarded into disinfectant solution. Using eight fresh, sterile tips the process was repeated by transferring 100µl from column 2 into column 3 (and 8 into 9). The process was continued until all wells in columns 6 and 12 contained 200µl. After mixing, 100µl was discarded from wells in columns 6 and 12 to waste. Finally, 100µl of prediluted bacterial culture (approx. 106 cfu/ml) was added, thus giving 200µl final volume in each well.

A blank plate was prepared for each set of eight samples in exactly the same way, except that 100µl of sterile 0.1% TSB was added instead of bacterial culture. This plate was used as the control plate against which the test plate(s) could be read. Test and control plates were sealed using autoclave tape and incubated for 18-30 hours at 37°C.

The microtitre plate reader (Model MRX, Dynatech Laboratories) was preset to gently agitate the plates and mix the contents. The absorbance at 540nm (hereinafter referred to for brevity and simplicity as " $A_{540}$ ") was used as a measure of turbidity resulting from bacterial growth. The control, un-inoculated plate for each set of samples was read first, and the plate reader then programmed to use the control readings to blank all other plate readings for the inoculated plates for the same set of test materials (i.e. removing turbidity due to perfume and possible colour changes during incubation). Thus, the corrected readings generated were absorbances resulting from turbidity from bacterial growth. The MIC was taken as the concentration of perfume/perfume component required to inhibit growth so that the change in absorbance during the incubation period was  $< 0.2 A_{540}$ .

#### Example 2: Steroid Biotransformation Assay

The ability of perfume components and mixtures of these components to inhibit the biotransformation of androstadienols to androstenones was determined *in vitro* using the method described below.

Corynebacterium sp. NCIMB 41018 (National Collections Of Industrial, Food and Marine Bacteria, 23 St Machar Drive, Aberdeen, AB24 3RY, Scotland, UK) (also known as Corynebacterium G41) was grown in 100ml of TSB supplemented with 0.1% w/v yeast extract (Oxoid) and 0.1% v/v Tween 80 (Sigma, Poole, UK) for 18-30 hours, in a shaken flask at 37°C. This culture was then harvested by centrifugation, and resuspended in 100 ml of biotransformation medium (consisting of a sterile semi-synthetic basal medium containing KH<sub>2</sub>PO<sub>4</sub> 1.6 g/l; (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> 5 g/l; Na<sub>2</sub>SO<sub>4</sub> 0.38 g/l; yeast nitrogen base 3.35 g/l; yeast extract 0.5 g/l; Tween 80 0.2 g/l; Triton X-100 0.2 g/l and MgCl<sub>2</sub>.6H<sub>2</sub>O 0.5 g/l).

Substrate androsta-5,16-dien-3 $\beta$ -ol (50mg/assay) was added to the bacterial suspension and incubated for 72 hours at 37°C with agitation (at 220-250rpm) in a 250 ml, baffled-Erlenmeyer flask.

Following biotransformation of androsta-5,16-dien-3 $\beta$ -ol to androst-16-en-3-one the bacteria were harvested and the cell pellet dried in air and then under vacuum.

The dried cells were then crushed and suspended in 100 ml of a mixture of diethyl ether, chloroform, ethanol, ethyl acetate and acetone (1:2:1:1:1 v/v, respectively), and stirred for 16 hours. The supernatant was then reduced to half its volume, filtered and evaporated at 30°C and 15 mmHg pressure. The resulting residue was re-dissolved in 5 ml AR grade methanol. Following sonication, the sample was analysed by HPLC on a Phenomenex Luna 5 micron, C18 reverse-phase HPLC column coupled to a Millipore-Waters 600E System Controller. Elute was passed through a Millipore-Waters 486 Tuneable absorbance detector and relative amounts of the steroid metabolite was determined by a Hewlett Packard HP 3396A Integrator printer. The composition of the HPLC mobile phase was aqueous methanol. The flow rate was 0.8 ml/min. Calibration curves were used to determine the molar quantities of pure steroid metabolites in biotransformed mixtures and hence the conversions.

Metabolites were analysed by HPLC-MS to determine their structure.

The biotransformation of androsta-5,16-dien-3 $\beta$ -ol to 5 $\alpha$ -androst-16-en-3-one by Corynebacterium NCIMB 41018 is as shown below:

It will be appreciated that the Coryneform bacteria used in Examples 1 and 2 are not the same strains. This is because the nutrient Tween-80 required for growth by Corynebacterium NCIMB 41018 (Example 2) is not suitable for inclusion in the growth medium used for MIC testing. As described above, during MIC testing, measurements are taken of the turbidity resulting from bacterial growth. Tween-80 when dissolved in an aqueous growth medium turns the medium cloudy. Thus, the addition of Tween-80 to a growth medium to be used for MIC testing would interfere with the readings, making an accurate determination of the turbidity due to bacterial growth impossible. Thus, a similar axillary Corynebacterium strain (C. xerosis NCTC 7243) is used in the MIC test, which does not require this nutrient for growth. The susceptibility of Corynebacterium xerosis NCTC 7243 to a variety of perfume components is likely to be very similar to that of Corynebacterium NCIMB 41018 as they are from the same genus.

### Example 3

Perfume A: Composition % by weight.

INGREDIENT	w/w%	
AGARBOIS (Q)	15	*
CINNAMIC ALCOHOL	. 2	
COUMARIN	1	
DIHYDROMYRCENOL	8	*
GERANIUM OIL	2	
HABANOLIDE (F)	3	
LILIAL (G)	10	
(4-ISOPROPYLCYCLOHEXYL)METHANOL	2	*
MEFROSOL (Q)	5	*
METHYL ANTHRANILATE	1	
METHYL CEDRYL KETONE	4	
METHYL DIHYDROJASMONATE (Q)	10	
PHENYL ETHYL ALCOHOL	15	
ROSACETONE	5	*
VANILLIN 5% IN DEP	17	
total	100.00%	

<sup>\*</sup> Materials of the invention

Trademarks: 'Q' = Quest International; 'F' = Firmenich; 'G' = Givaudan

Perfume B: Composition % by weight.

INGREDIENT	w/w	%
ACETYL CEDRENE	7.5	:k
AGARBOIS (Q)	. 6	*
ALDEHYDE MNA 10% DEP	1	
ALLYL AMYL GLYCOLATE (Q)	2.2	*
AMBER CORE (Q)	0.5	
ARMOISE TUNISIAN	0.4	*
BANGALOL (Q)	0.5	
BENZYL SALICYLATE (Q)	8.5	

BERGAMOT OIL	7.5	
BOURGEONAL (Q)	0.5	
CARVONE LAEVO (Q) 10% DEP	1	
CEDARWOOD VIRGINIAN OIL	1.1	
cis-3-HEXENYL SALICYLATE	1.5	
CISTULATE (Q) 10% DEP	2	
CORIANDER	0.3	
COUMARIN	0.6	-
CYCLOHEXYLOXYACETIC ACID, ALLYL ESTER	0.2	
CYCLOPENTADECANOLIDE	2.2	
DIHYDROMYRCENOL (Q)	13	*
ETHYLENE BRASSYLATE	1.5	
GERANIUM OIL	1.4	-
HELIONAL	0.3	
HEXYL CINNAMIC ALDEHYDE	2.5	
IONONE (Q)	1.5	
ISO AMBOIS (Q)	7.5	
ISO BORNYL ACETATE	0.6	*
ISOBORNYL CYCLOHEXANOL	1.5	
LAVANDIN OIL	0.3	
LILIAL (G)	6.8	
METHYL CHAVICOL	1.2	
METHYL DIHYDROJASMONATE SUPER (Q)	6.4	
MOSS OAKMOSS SYNTHETIC	0.2	
NUTMEG PURE	0.2	
PEPPERMINT CHINESE 10% DEP	3.5	*
PETITGRAIN PARAGUAY	0.2	
ROSE OXIDE RACEMIC 10% DEP	0.5	
STYRALLYL ACETATE	0.4	
TERPINEOL ALPHA	2.5	*
TETRAHYDROLINALOL	4.5	
total	100.0	00%

<sup>\*</sup> Materials of the invention

# Perfume C: Composition % by weight.

INGREDIENT	w/w%	
ACETYL CEDRENE (Q)	7 *	
AGARBOIS (Q)	15 *	•
ALDEHYDE MNA 10% DEP	2.5	
BENZYL SALICYLATE (Q)	6.4	

cis-JASMONE	1.2	*
CITRONELLAL	2.2	*
COUMARIN	1.3	
CYCLOPENTADECANOLIDE	6.6	
DIHYDROMYRCENOL (Q)	8.5	*
ETHYLENE BRASSYLATE	2.3	
HEXYL CINNAMIC ALDEHYDE	3.5	
ISO AMBOIS (Q)	7	
ISO BORNYL ACETATE	2.6	*
LILIAL (G)	5.4	
MARENIL (Q)	1.3	*
MEFROSOL (Q)	5.4	*
METHYL DIHYDROJASMONATE SUPER (Q)	7.6	
PETITGRAIN PARAGUAY	1.2	
TERPINEOL ALPHA	3	*
TETRAHYDROGERANIOL	10	*
total	100.0	0%

<sup>\*</sup> Materials of the invention

## Perfume D: Composition % by weight.

INGREDIENT	w/w%	
4 (5 ETHYLDICYCLOI2 2 THERTYL 2) CVCLOUEYANOL	1 2	*
4-(5-ETHYLBICYCLO[2.2.1]HEPTYL-2)-CYCLOHEXANOL	5.3	*
ACETYL CEDRENE (Q)		
ALDEHYDE C11 (UNDECYLENIC ALDEHYDE) 10% DEP		
ALDEHYDE MNA 10% DEP	8.0	_
ALLYL AMYL GLYCOLATE (Q)	1.3	*
ARMOISE TUNISIAN	0.2	*
BANGALOL (Q)	0.3	
BENZYL SALICYLATE (Q)	5.1	
BERGAMOT OIL	4.8	
CEDARWOOD VIRGINIAN OIL	1.1	
CITRONELLAL	2	*
CITRONELLOL	6.9	
CYCLOPENTADECANOLIDE	2.3	
DIHYDROMYRCENOL (Q)	15.8	*
ETHYLENE BRASSYLATE	8.8	
FENCHYL ACETATE	2.5	*
HEXYL CINNAMIC ALDEHYDE	5.1	
IONONE (Q)	3.5	
ISOBORNYL CYCLOHEXANOL	1.8	
METHYL DIHYDROJASMONATE SUPER (Q)	5.5	

PARA TERT BUTYL CYCLOHEXYL ACETATE	3.4	•
PARADISAMIDE (Q)	2.8	*
PEPPERMINT CHINESE 10% DEP	4.3	*
PHENYLETHYL ALCOHOL	6	
ROSE OXIDE RACEMIC 10% DEP	2.1	
ROSEACETONE	3.7	*
TETRAHYDROGERANIOL	2	*
total	100.00%	

<sup>\*</sup> Materials of the invention

## Perfume E: Composition % by weight.

INGREDIENT	w/w%	ó
4-(5-ETHYLBICYCLO[2.2.1]HEPTYL-2)-CYCLOHEXANOL	2.3	*
AGARBOIS (Q)	4	*
ALDEHYDE C11 (UNDECYLENIC ALDEHYDE) 10% DEP	1.2	
AMBER CORE (Q)	4.3	
CARVONE LAEVO (Q) 10% DEP	3.8	
CEDARWOOD VIRGINIAN OIL	1.8	
cis-JASMONE	0.5	*
CISTULATE (Q) 10% DEP	0.9	
CITRONELLOL	3.6	
CORIANDER	0.2	
COUMARIN	0.9	
DIHYDROMYRCENOL (Q)	4.5	*
ETHYLENE BRASSYLATE	6.2	
FENCHYL ACETATE	3.6	*
HEXYL CINNAMIC ALDEHYDE	6.8	
HEXYL SALICYLATE	7.5	
LILIAL (G)	6.5	•
MARENIL (Q)	2.6	*
METHYL CHAVICOL	0.4	
METHYL DIHYDROJASMONATE SUPER (Q)	3.5	
METHYL OCTYL ACETALDEHYDE 10% DEP	5.5	*
MOSS OAKMOSS SYNTHETIC	0.2	
PEPPERMINT CHINESE 10% DEP	3.4	*
PETITGRAIN PARAGUAY	2.1	
PHENYLETHYL ALCOHOL	7.1	
TERPINEOL ALPHA	6.4	*
TETRAHYDROGERANIOL	8.2	*
TETRAHYDROLINALOL	2	



100.00%

total

\* Materials of the invention

### Example 4: Product Base Examples

The following are typical formulations of deodorant products which comprise a perfume composition in accordance with the invention. These formulations are made by methods common in the art.

#### 1. Deodorant Sticks

Ingredient	Content (% by weight)		
	Formulation 1A	Formulation 1B	
Ethanol		8.0	
Sodium Stearate	7.0	6.0	
Propylene glycol	70.0	12.0	
Perfume	1.5	2.0	
PPG-3 Myristyl ether	ſ	28.0	
PPG-10 Cetyl ether		10.0	
Cyclomethicone		34.0	
Water	21.5		
	·	·	

### 2. Aerosols

Ingredient

Content % by weight

Formulation 2B Formulation 2A

up to 100. Ethanol B

Propylene glycol

as required

Ingredient

Perfume	2.0	1.2
Chlorhydrol microdry		31.8
Silicone Fluid DC344		up to 100
Bentone gel IPP	•	13.65
Dimethyl ether	20.0	
Concentrate		22.0
Water	23.0	

Ethanol (Denatured) up to 100

Perfume 1.0

DC345 Fluid<sup>(i)</sup> 15.0

Hydrocarbon Propellant, 30 psig<sup>(ii)</sup> 60.0

(i) DC345 fluid (INCI name - CYCLOPENTA-SILOXANE) is a volatile, low viscosity, silicone fluid. It is non-greasy providing a light, silky feel on the skin.

Content % by weight

(ii) The hydrocarbon propellant can be any deodorised blend of n-butane, n-propane and isobutane having a pressure of 30 pounds per square inch gauge or 2.109 kg/cm<sup>2</sup> gauge (308 kPa).

#### 3. Roll ons

Ingredient	Content % by weight	
	Formulation 3A	Formulation 3B
Ethanol	to 100%	60.0
Klucel MF		0.65

Cremphor RM410		0.5
Perfume	0.5	1.0
AZTC*	20.0	
Cyclomethicone	68.0	
Dimethicone	5.0	
Silica	2.5	
Water		37.85

<sup>\*</sup> Aluminium zirconium tetrachlorohydro glycinate

Perfume compositions A to E embodying this invention (see Example 3 above) were prepared and tested for deodorant action in underarm products, particularly an aerosol product of Formulation 2C, using an Odour Reduction Value test generally as described in US 4,278,658.

The Odour Reduction Value test was carried out using a panel of 40 Caucasian male subjects. A standard quantity (approximately 1.75g) of an aerosol product containing one of the perfume compositions or an unperfumed control was applied to the axillae of the panel members in accordance with a statistical design.

After a period of five hours, the underarm odour was judged by three trained female assessors who scored the odour intensity in accordance with a 0 to 5 scale, as shown below:

Score	Odour level	Conc. of aqueous isovaleric acid (ml/I)
0	No odour	0
1	Slight	0.013
2	Definite	0.053
3	Moderate	0.22
4	Strong	0.87

5

26

Very Strong

3.57

Average scores for each test product and the control product were then determined. The score for each test product was subtracted from the score for the control product and the reduction expressed as a percentage to give the Odour Reduction Value(%).

Perfume compositions A to E were all found to exhibit significant deodorant activity.

For example, Perfume A contains 35% of perfume components of the invention. Excluding diluents, this percentage increases to 42.2%. For this perfume, present at 1.0% in an aerosol product of Formulation 2C above, the Odour Reduction Value(%) compared to an unperfumed control was 48.3% (5 hours).

The Odour Reduction Value (%) compared to an unperfumed control for Perfume B was 44.6% (5 hours), for Perfume C 35.3% (5 hours) and for Perfume E 28.2% (5 hours).

#### CLAIMS

- 1. A perfume composition comprising at least 30% by weight of one or more of the following perfume components; Armoise Tunisian oil, para-tert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, 2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, dihydromyrcenol, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, alpha-terpineol, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal.
- 2. A perfume composition comprising at least 3 of the following perfume components; Armoise Tunisian oil, para-tert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, 2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, dihydromyrcenol, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, alpha-terpineol, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal.
- 3. A perfume composition according to claim 2, wherein the perfume composition comprises at least 30% by weight of at least 3 of the specified perfume components.

- 4. A deodorant product comprising a perfume composition according to any one of claims 1, 2 or 3.
- 5. Use of one or more of the following perfume components; Armoise Tunisian oil, paratert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, carboxylate, 2,2,2-trichloro-1-3-methyl-5-phenylpentan-1-ol, isopropylcyclohexyl)methanol, allyl amyl glycolate, acetyl cedrene, isobornyl acetate, phenylethyl acetate, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal, as a deodorant active.
- 6. Use of one or more of the following perfume components; Armoise Tunisian oil, paratert butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4ethyltricyclo[5.2.1.0{2,6}]decane-2-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, (4-2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, carboxylate, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1isopropylcyclohexyl)methanol, acetyl phenylethyl acetate, isobornyl acetate. allyl amyl glycolate, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal, in the manufacture of a composition for reducing or preventing body malodour.
- 7. A method for reducing or preventing body malodour by topically applying to human skin a composition comprising a perfume component selected from at least one of the following; Armoise Tunisian oil, para-tert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, 2-ethyl-N-methyl-N-(3-



methylphenyl)butanamide, dihydromyrcenol, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, alpha-terpineol, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal.

- 8. A method according to claim 7 wherein the perfume component is selected from at least one of the following; Armoise Tunisian oil, para-tert.butylphenylacetonitrile, dihydrolinalol, N-ethyl-N-(3-methylphenyl)propionamide, 4-(5-ethylbicyclo[2.2.1]heptyl-2)-cyclohexanol, ethyltricyclo[5.2.1.0{2,6}]decane-2-carboxylate, 2-ethyl-N-methyl-N-(3-methylphenyl)butanamide, (4-isopropylcyclohexyl)methanol, 3-methyl-5-phenylpentan-1-ol, 2,2,2-trichloro-1-phenylethyl acetate, isobornyl acetate, allyl amyl glycolate, acetyl cedrene, tetrahydrogeraniol, citronellal, cuminic aldehyde, 1,3,3-trimethyl-2-norbornanyl acetate, cis-jasmone, methyl octyl acetaldehyde, gamma-octalactone, octyl acetate, pine American oil, peppermint (Chinese), 1,3,3-trimethyl-2-norbornanol, gamma-nonalactone, octahydro-2H-chromen-2-one, cis-4-decenal, 3-(3-isopropylphenyl)butanal.
- 9. A method according to claim 7 or 8, wherein the composition is a perfume composition.
- 10. A method according to claim 9, wherein the perfume composition comprises at least 30% by weight of at least one of the specified perfume components.

### INTERNATIONAL SEARCH REPORT

Internatic plication No PCT/3/03083

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 C11B9/00 A61Q13/00

A61Q15/00

According to International Patent Classification (IPC) or to both national classification and IPC

#### **B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols) IPC 7 C11B A61Q A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ, CHEM ABS Data

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Section Ch, Week 199430 Derwent Publications Ltd., London, GB; Class D21, AN 1994-245645 XP002027043 & JP 06 179610 A (SHISEIDO CO LTD), 28 June 1994 (1994-06-28) abstract	1-10
X	WO 96 04940 A (PROCTER & GAMBLE) 22 February 1996 (1996-02-22) page 3, line 5 -page 7, line 2 claim 1	1-3

Further documents are listed in the continuation of box C.	χ Patent family members are listed in annex.
Special categories of cited documents:      A' document defining the general state of the art which is not considered to be of particular relevance      E' earlier document but published on or after the international filling date      L' document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)      O' document referring to an oral disclosure, use, exhibition or other means      P' document published prior to the international filing date but later than the priority date claimed	<ul> <li>"T" later document published after the International filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</li> <li>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</li> <li>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</li> <li>"&amp;" document member of the same patent family</li> </ul>
Date of the actual completion of the international search  9 October 2003	Date of malling of the international search report 23/10/2003
Name and mailing address of the ISA  European Patent Office, P.B. 5818 Patentiaan 2  NL – 2280 HV Rijswijk  Tel. (+31–70) 340–2040, Tx. 31 651 epo nl,  Fax: (+31–70) 340–3016	Authorized officer  Dekeirel, M

### INTERNATIONAL SEARCH REPORT

PCT/6 B/03083

College   Challen of document, with indication, where appropriate, of the retornal passages	C.(Continua	etion) DOCUMENTS CONSIDERED TO BE RELEVANT	101/6 3/03083
PIDDOCK CHRISTOPHER CHARLES (2B);   CLEMENTS) 11 November 1999 (1999-11-11)     page 9, line 6 -page 12, line 14     claims 1,3	Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
(NL)) 23 June 1994 (1994-06-23) example 2  WO 02 39971 A (COLGATE PALMOLIVE CO) 23 May 2002 (2002-05-23) claims 1-4  X	X	;PIDDOCK CHRISTOPHER CHARLES (GB); CLEMENTS) 11 November 1999 (1999-11-11) page 9, line 6 -page 12, line 14	1-3
X	X	(NL)) 23 June 1994 (1994-06-23)	1-3
STANLEY (GB)) 3 October 1996 (1996-10-03) page 2, line 10 - line 37 page 4, line 4 - line 34 page 5, line 24 - line 25 claim 1  X WO 02 49600 A (BEHAN JOHN MARTIN ; PERRING KEITH DOUGLAS (GB); MCNULTY DAVID (GB);) 27 June 2002 (2002-06-27) examples 1-6,10  X WO 99 44575 A (COLGATE PALMOLIVE CO) 10 September 1999 (1999-09-10) claims 1-6,11-13  X EP 1 029 841 A (GIVAUDAN ROURE INT) 23 August 2000 (2000-08-23) page 3, line 43 - line 50 example 4 claims 1,2,4,5  X EP 1 113 105 A (INT FLAVORS & FRAGRANCES INC) 4 July 2001 (2001-07-04) examples A,B claims 1,7  X WO 01 24769 A (BRETLER GIL ; FIRMENICH & CIE (CH)) 12 April 2001 (2001-04-12) claims 1-7  X WO 00 01361 A (BEHAN JOHN MARTIN ; PERRING KEITH DOUGLAS (GB); QUEST INT (NL)) 13 January 2000 (2000-01-13) page 6, Table 2 page 4, line 5 - line 10 claims 1,6-12	Х	23 May 2002 (2002-05-23)	1,4-9
KEITH DOUGLAS (GB); MCNULTY DAVID (GB);   27 June 2002 (2002-06-27)   examples 1-6,10	x	STANLEY (GB)) 3 October 1996 (1996-10-03) page 2, line 10 - line 37 page 4, line 4 - line 34 page 5, line 24 - line 25	1,4-10
10 September 1999 (1999-09-10) claims 1-6,11-13  X EP 1 029 841 A (GIVAUDAN ROURE INT) 23 August 2000 (2000-08-23) page 3, line 43 - line 50 example 4 claims 1,2,4,5  X EP 1 113 105 A (INT FLAVORS & FRAGRANCES INC) 4 July 2001 (2001-07-04) examples A,B claims 1,7  X W0 01 24769 A (BRETLER GIL ;FIRMENICH & CIE (CH)) 12 April 2001 (2001-04-12) claims 1-7  X W0 00 01361 A (BEHAN JOHN MARTIN ;PERRING KEITH DOUGLAS (GB); QUEST INT (NL)) 13 January 2000 (2000-01-13) page 6, Table 2 page 4, line 5 - line 10 claims 1,6-12	x	KEITH DOUGLAS (GB); MCNULTY DAVID (GB);) 27 June 2002 (2002-06-27)	2
23 August 2000 (2000-08-23) page 3, line 43 - line 50 example 4 claims 1,2,4,5  X EP 1 113 105 A (INT FLAVORS & FRAGRANCES INC) 4 July 2001 (2001-07-04) examples A,B claims 1,7  X W0 01 24769 A (BRETLER GIL ;FIRMENICH & CIE (CH)) 12 April 2001 (2001-04-12) claims 1-7  X W0 00 01361 A (BEHAN JOHN MARTIN ;PERRING KEITH DOUGLAS (GB); QUEST INT (NL)) 13 January 2000 (2000-01-13) page 6, Table 2 page 4, line 5 - line 10 claims 1,6-12	x	10 September 1999 (1999-09-10)	2,4-9
INC) 4 July 2001 (2001-07-04) examples A,B claims 1,7  X W0 01 24769 A (BRETLER GIL ;FIRMENICH & 1,4-10 CIE (CH)) 12 April 2001 (2001-04-12) claims 1-7  X W0 00 01361 A (BEHAN JOHN MARTIN ;PERRING KEITH DOUGLAS (GB); QUEST INT (NL)) 13 January 2000 (2000-01-13) page 6, Table 2 page 4, line 5 - line 10 claims 1,6-12	X	23 August 2000 (2000-08-23) page 3, line 43 - line 50 example 4	2
CIE (CH)) 12 April 2001 (2001-04-12) claims 1-7  WO 00 01361 A (BEHAN JOHN MARTIN ; PERRING KEITH DOUGLAS (GB); QUEST INT (NL)) 13 January 2000 (2000-01-13) page 6, Table 2 page 4, line 5 - line 10 claims 1,6-12	x	INC) 4 July 2001 (2001-07-04) examples A,B	1,6-10
KEITH DOUGLAS (GB); QUEST INT (NL)) 13 January 2000 (2000-01-13) page 6, Table 2 page 4, line 5 - line 10 claims 1,6-12	x	CIE (CH)) 12 April 2001 (2001-04-12)	1,4-10
-/	X	KEITH DOUGLAS (GB); QUEST INT (NL)) 13 January 2000 (2000-01-13) page 6, Table 2 page 4, line 5 - line 10	5–19
		-/	
·			



PCT/G. Plication No

Relevant to claim No.  5–9
5-9
1
1,4-10

## INTERNATIONAL EARCH REPORT

Internation No PCT/4 3/03083

Patent document cited in search report		Publication date		Patent family member(s)	Publication date
JP 6179610	A	28-06-1994	JP	3024865 B2	27-03-2000
WO 9604940	A	22-02-1996	USU AU BR CA CDE EP EP PRU USS USS US US	5939060 A 3242995 A 9508569 A 2196702 A1 2330720 A1 9700401 A3 29522186 U1 1002549 A1 1232761 A1 0774980 A1 10503958 T 2002069840 A 318553 A1 2149025 C1 9604940 A1 6146621 A 5663134 A 5783544 A 2003005522 A1 6248135 B1 5670475 A	17-08-1999 07-03-1996 23-12-1997 22-02-1996 22-02-1996 13-08-1997 15-06-2000 24-05-2000 21-08-2002 28-05-1997 14-04-1998 08-03-2002 23-06-1997 20-05-2000 22-02-1996 14-11-2000 02-09-1997 21-07-1998 09-01-2003 19-06-2001 23-09-1997
 WO 9957233	Α	11–11–1999	US US  AU WO	2001044392 A1 6077318 A 	22-11-2001 20-06-2000  23-11-1999 11-11-1999
WO 9413766	A	23-06-1994	AT AU BR DE WO EP ES JP JP JP US ZA	140723 T 5811294 A 9307615 A 69303830 D1 9413766 A2 0673408 A1 2089913 T3 2839881 B2 10245584 A 2933719 B2 5698253 A 9309286 A	15-08-1996 04-07-1994 15-06-1999 29-08-1996 23-06-1994 27-09-1995 01-10-1996 16-12-1998 14-09-1998 16-08-1999 16-12-1997 12-06-1995
WO 0239971	A	23-05-2002	AU WO	2889902 A 0239971 A2	27-05-2002 23-05-2002
WO 9630470	A	03-10-1996	AU AU BR DE WO EP ES JP US	700662 B2 5109296 A 9607764 A 69613823 D1 69613823 T2 9630470 A1 0819161 A1 2160235 T3 11506475 T 5856295 A	14-01-1999 16-10-1996 19-01-1999 16-08-2001 22-11-2001 03-10-1996 21-01-1998 01-11-2001 08-06-1999 05-01-1999
WO 0249600	Α	27-06-2002	AU EP	1620402 A 1343466 A1	01-07-2002 17-09-2003

## INTERNATIONAL\_SEARCH REPORT

Internati \_\_\_\_\_\_ pplication No PCT/G \_\_\_\_\_\_/03083

		1				101/G	7 03003
	tent document in search report		Publication date		Patent family member(s)		Publication date
WO	0249600	Α		WO	0249600	A1	27-06-2002
WO	9944575	Α	10-09-1999	US	6180121	B1	30-01-2001
				AU	749473	B2	27-06-2002
				AU	3311999	Α	20-09-1999
				BR	9908579	Α	21-11-2000
				CA	2322284	A1	10-09-1999
				EP	1061894	A1	27-12-2000
				HU	0101149	A2	28-08-2001
				JP	2002505264	· T	19-02-2002
				NO	20004391	. A	03-11-2000
				PL	342653	A1	02-07-2001
				WO	9944575	A1	10-09-1999
				ZA	9901768	A	11-10-2000
EP	1029841	Α	23-08-2000	EP	1029841		23-08-2000
				ΑT	236865		15-04-2003
				AU	1760100		24-08-2000
				BR	0000850		21-08-2001
				CN	1266838		20-09-2000
				DE	60001997		15-05-2003
				JP	2000239691		05-09-2000
				SG	83775		16-10-2001
				US	6297211		02-10-2001
				ZA	200000724	Α	21-05-2000
EP	1113105	Α	04-07-2001	US	6379658		30-04-2002
				BR	0001834		11-09-2001
				EP	1113105 	A2	04-07-2001
WO	0124769	Α	12-04-2001	BR	0014476		04-06-2002
				EP	1221934		17-07-2002
				MO	0124769		12-04-2001
	•			JP	2003510343		18-03-2003
				US 	2002142364	A1	03-10-2002
WO	0001361	Α	13-01-2000	AU	4636999		24-01-2000
				EP	1093358		25-04-2001
				WO	0001361 	. Al	13-01-2000
WO	0001352	Α	13-01-2000	AU	4630599		24-01-2000
				BR	9911897		27-03-2001
	•			EP	1094784		02-05-2001
				WO	0001352		13-01-2000
				JP 	2002519367 	′ T 	02-07-2002
JP	2000355696	Α	26-12-2000	NONE			
	03000648	Α	03-01-2003	WO	03000648	λ1	03-01-2003